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Mechanical properties of MgB_2 bulk fabricated by Spark Plasma Sintering

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Abstract

A clear understanding of mechanical properties of superconducting bulk materials is required for their reliable integration together with other materials constituting the superconducting devices. In this study, evaluations of mechanical properties of a high packing ratio MgB_2 bulk processed by Spark Plasma Sintering-SPS were carried out. Mechanical properties of the high packing ratio MgB_2 bulk processed by SPS were improved than those of MgB_2 bulks processed by conventional route. The improvement of the mechanical properties with increasing the packing ratio could be approximated by using exponential equations.

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Keywords: MgB_2 bulk; Spark plasma sintering; Bending test; Mechanical properties

1. Introduction

Since grain boundaries of MgB_2 superconducting bulk are not weak links, MgB_2 bulk can be processed by just sintering without crystal growth [1-3]. Thus, it is not so difficult to obtain large MgB_2 bulk which shows uniform distribution of trapped magnetic field. On the other hand, $REBa_2Cu_3O_x$ (RE123, RE: Y or rare-earth elements.) bulk has to be a single-grain, which is processed by melt-processing using a seed crystal. It is difficult to obtain large single-grain RE123 bulk, due to the undesirable nucleation at the region apart from the seed crystal. Since superconducting bulk materials are subjected to electromagnetic force and thermal stress in the superconducting devices [4-6], a clear understanding of mechanical behaviour of superconducting bulk materials is required for their reliable integration together with other materials constituting the superconducting devices. However, mechanical properties of MgB_2 bulk have not been understood extensively in comparison with those of RE123 bulk. In the previous study, evaluations of mechanical properties have been carried out for MgB_2 bulks processed by capsule method and Hot Isostatic Pressing-HIP through bending tests for specimens cut from these bulks [7]. HIP is effective in improving the packing ratio. Mechanical properties of the MgB_2 bulks were improved with increasing the packing ratio [7]. Fracture strength of the

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MgB_2 bulk processed by capsule method was lower than those of RE123 bulks [7]. On the other hand, fracture strength of the improved MgB_2 bulk processed by HIP was higher than those of RE123 bulks [7]. It is known that Spark Plasma Sintering-SPS process is also effective in improving the packing ratio [8,9]. Thus enhance the samples density. In this study, MgB_2 bulk was processed by SPS. Mechanical properties of the SPS bulk were evaluated through bending tests of specimens cut form the bulk. Mechanical properties of the SPS bulk were compared with those of other MgB_2 bulks processed by capsule method and HIP reported in Ref. [7].

2. Experimental procedures

2.1. Sample preparation

Grade A magnesium diboride from ABCR GmbH (Karlsruhe, Germany) was used as the starting powder. The powder was loaded into a graphite die and processed using the Spark Plasma Sintering system (FCT System GmbH, HD25, Rauenstein, Germany) in DC mode. The pulsed electric current (2000 A, 4 V) was passed through the sample under dynamic vacuum (10^{-3} bar) while a 50 MPa uniaxial pressure was applied. During the experiment, the temperature (1100°C, during 30 min), applied pressure and displacement or shrinkage of the sample was recorded. Fig. 1 presents the displacement and speed of the punches in the thickness direction showing the sample shrinkage during spark plasma sintering experiment of the sample powder. The as-prepared discs (Fig. 2) were polished before characterization to remove the graphite coating deposited on the samples during the process. The determination of the packing ratio of the sample was performed using the Archimedes method with Ethanol. The MgB_2 bulk with 30 mm diameter and 9 mm thick processed is denser (98% relative density of the theoretical value) than the samples obtained by conventional sintering route as mentioned in the followings. Superconducting properties of MgB_2 bulk processed by SPS are reported in Ref. [10].

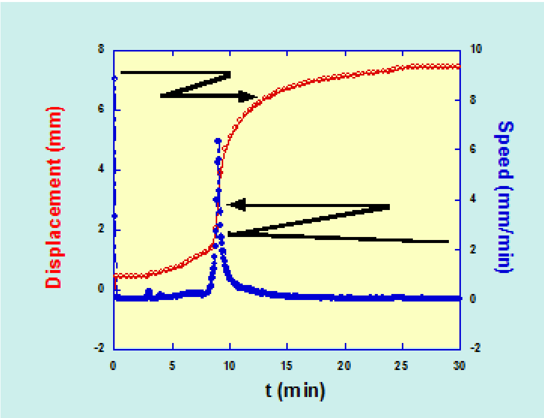

Fig. 1. The displacement and the punches speed showing the sample shrinkage during spark plasma sintering experiment (1100°C, 50 MPa) of the sample powder.

Fig. 2. As-processed MgB_2 sample.

2.2. Evaluation of mechanical properties

Mechanical properties of the MgB_2 bulk processed by SPS were evaluated through bending tests of specimens cut from the bulk. Bending test specimens with the dimensions of around 2.8 x 2.1 x 24 mm^3 were cut from the bulk sample by using a wire discharge cutting machine. The longitudinal direction of the specimens was almost perpendicular to the thickness direction of the bulk. After that, one strain gage with the gage length of 0.2 mm was glued to the centre of the 2.8 x 24 mm^2 surface of the specimens. Four-point bending load was applied at room temperature in the 2.1 mm direction of the specimens under the crosshead speed of 0.2 mm/min. Stress σ caused by the four-point bending load was calculated by the following equation.

$$\sigma = \frac{3P(L-l)}{2wt^2} \qquad (1)$$

Where P is the applied load, L is outer supporting span (21 mm), l is upper loading span (7 mm), w is width (2.8 mm) and t is thickness (2.1 mm) of the specimens.

3. Results and discussion

Fig. 3 presents stress-strain curves of specimens cut from the MgB_2 bulk processed by SPS. Data points represent the fracture points of the specimens. Data points of other MgB_2 bulks reported in Ref. [7] are also shown for reference. Bending tests of other MgB_2 bulks were carried out under the same conditions as those of the bending tests in the present study. The packing ratio of sintered materials, such as MgB_2 bulk, commonly depends on sintering method. MgB_2 bulk is usually processed by capsule method [1]. Capsule method is performed under the ambient pressure. The packing ratio of an MgB_2 bulk processed by capsule method is around 50% [7]. On the other hand, the packing ratio of MgB_2 bulk was improved by HIP up to around 92% [7]. However, the packing ratio of an MgB_2 bulk processed by HIP after the capsule method is not so high (63%) [7]. Further improvement of the packing ratio is observed for the MgB_2 bulk processed by SPS in the present study. The minimum, average and maximum fracture strength values of the SPS bulk were 223, 315 and 450 MPa, respectively. Fracture strength of the high packing ratio MgB_2 bulk processed by SPS is higher than those of other MgB_2 bulks. The improvement of the fracture strength is attributable to the increase of the net cross-sectional area which suppresses the deformation. Reduction of defects, where the stress concentration occurs, with increasing the packing ratio is another reason for the improvement of the fracture strength. Stress-strain curves of the specimens cut from the SPS bulk are almost linear until the fracture. Stress-strain curves of the HIP bulk are also linear as reported in Ref. [7]. The linear stress-strain behavior represents that these MgB_2 bulks are brittle.

Fig. 4 presents Weibull plots of the fracture strength of specimens cut from the MgB_2 bulk processed by SPS. Weibull plots are used for the evaluation of scatter of the fracture strength data [11]. Slope obtained by linear fitting of the plots represents Weibull coefficient; larger Weibull coefficient value means smaller scatter of the fracture strength data. The Weibull coefficient value of the SPS bulk is 4.5, which is smaller than the value of RE123 bulks 8-10 reported in Ref. [12].

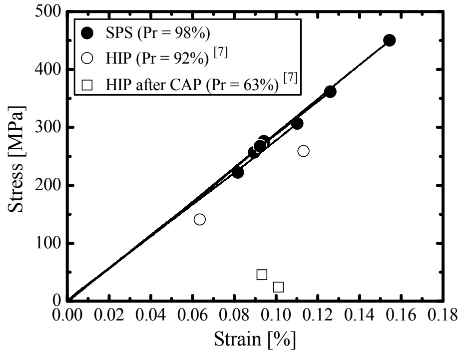

Fig. 3. Stress-strain curves of specimens cut from MgB_2 bulk processed by Spark Plasma Sintering-SPS. HIP, CAP and Pr represent hot isostatic pressing, capsule method and packing ratio, respectively.

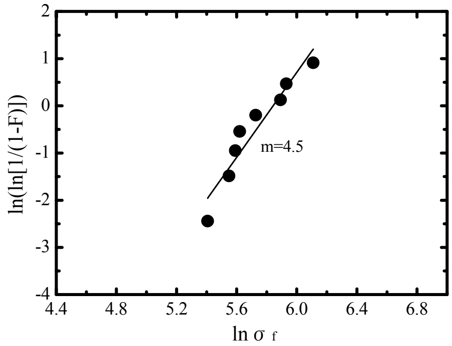

Fig. 4. Weibull plots of fracture strength of specimens cut from MgB_2 bulk processed by SPS. σ_f, F and m represent fracture strength, cumulative fracture probability and Weibull coefficient, respectively.

Fig. 5 presents relationship between the average fracture strength value and the packing ratio of the MgB_2 bulks. The fracture strength was improved with increasing the packing ratio. It has been reported that relationship between the fracture strength σ_f and porosity p of ceramic materials can be expressed by using an exponential equation [13].

$$\sigma_f = \sigma_0 \exp(-bp) \qquad (2)$$

Where σ_0 is the fracture strength at the porosity of 0 % and b is constant. In the previous study, relationship between the fracture strength and the packing ratio of the MgB_2 bulks processed by HIP (Pr=92%), HIP after capsule method (Pr=63%) and just capsule method (Pr=50%) could be approximated by using an exponential equation [7]. Solid line in Fig. 5 represents the approximation of the data of these MgB_2 bulks with the packing ratio of 50-92%. Relationship between the fracture strength σ_f and the packing ratio Pr of the MgB_2 bulks could be expressed by the following equation [7].

$$\sigma_f = 0.772 \exp(0.0612\ \mathrm{Pr}) \qquad (3)$$

By using Eq. (3), the average fracture strength value of the MgB_2 bulk processed by SPS (Pr=98%) is estimated to be 311 MPa, which is close to the average fracture strength value obtained in the present study (315 MPa). Data point of the SPS bulk is almost on the approximation curve (see Fig. 5).

Young's modulus was evaluated from the slope of the stress-strain curves of the specimens. Fig. 6 presents relationship between the average Young's modulus value and the packing ratio of the MgB_2 bulks. Young's modulus was also improved with increasing the packing ratio. Relationship between the average Young's modulus value and the packing ratio could be approximated by using an exponential equation as shown in Fig. 6. The relationship could be expressed by the following equation.

$$E = 1.808 \exp(0.0518 \text{ Pr}) \qquad (4)$$

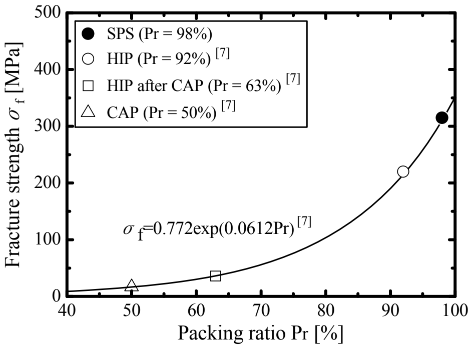

Fig. 5. Relationship between average fracture strength value and packing ratio of MgB_2 bulks.

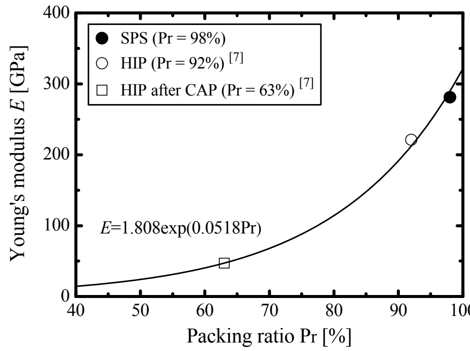

Fig. 6. Relationship between average Young's modulus value and packing ratio of MgB_2 bulks.

4. Conclusion

In order to evaluate mechanical properties of a high packing ratio MgB_2 bulk processed by Spark Plasma Sintering-SPS, four-point bending tests of specimens cut from the bulk sample were carried out at room temperature. Mechanical properties of the SPS bulk are compared with those of other MgB_2 bulks processed by conventional route. The mechanical properties, such as fracture strength and Young's modulus, of the high packing ratio MgB_2 bulk processed by SPS were improved. The improvement of the mechanical properties with increasing the packing ratio could be expressed by using exponential equations.

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